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FRENCH BAUXITE PROSPECTION MISSION IN THE LOWER MEKONG BASIN

NOTE ON BAUXITE ANALYSES

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Chief of Mission

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LABORATORY WORK

1. Preparation of samples

Laterite samples are usually sent from the field in cloth bags (approximately 1 to 2 kg per bag). Field operators should fix a consignment label to each bag, so that the numbers of the samples may be conveniently inscribed in a reception register.

The samples are often wet. Therefore, before crushing, they must often be dried by placing the bags in a drying oven with a temperature not exceeding 120° centigrade. During the dry season, when the samples have been sufficiently dried in the sun, it is not necessary to place the bags in the drying oven.

After drying, the samples are crushed, ground and screened in a 100 mesh screen. If the grinding apparatus is well adjusted, a single operation should be sufficient. Any oversize will be held by the screen.

Part of the ground sample, say 100 to 150 g. is to be placed in cardboard or plastic boxes for storage. A label is placed in each box indicating the number of the sample as well as any other useful data such as the place and condition of sampling and the depth at which the sample was taken.

Another part of the ground sample, 10 to 20 g, is placed in a 50 cc beaker (with a number corresponding to the sample number) for chemical laboratory analyses.

/2. Drying of

2. Drying of samples

In the chemical laboratory, the samples in the 50 cc beakers are stoved in a drying oven during one hour at a temperature of 110° to 120° centigrade; the beakers are then removed from the oven, closed and left to cool in a dessicator during three hours, protected from humidity.

All weight determinations are done by a simple weighing operation.

3. Physical analysis - Determination of ignition loss

After the dried sample has cooled, 1 gram of powder is weighed in a covered, numbered, calcinated and tared silica crucible. The numbers of the crucibles, both full and empty should be listed down in special registers.

The crucibles are then placed in an oven heated at 1,000° centigrade, which temperature is maintained for one hour.

As soon as heating is discontinued, the crucibles are removed and placed to cool for two hours in a dessicator; they are then weighed.

We have:

$P_1 - P_2 \times 100 = H_2O$ %

4. Chemical analyses

While one gram of powder is being weighed for ignition loss, another gram of powder is also weighed for chemical analyses.

(a) Determination of total silica content - Three-acids method

Reaction solution:

485 cc H_2O
115 cc SO_4H_2
100 cc NO_3H
300 cc ClH

The second gram of powder is then placed in a 250 cc pyrex beaker, covered by a watch glass. The beaker will receive a label showing the sample number.

/This powder

This powder is then attacked by about 75 cc of the above-mentioned mixture of sulfuric, hydrochloric and nitric acid and water during a period of one, two or three hours according to the rate of reaction of the various samples, on a sand bath at a temperature of approximately 450° centigrade. The whole is then boiled until all elements become soluble apart from the silica and the reaction residue produces thick white fumes. After ten more minutes, the reaction may be considered completed.

The result is then withdrawn from the fire and left to cool; 80 to 100 cc of acidulated water is added (water containing 10 cc of hydrochloric acid per 1,000), and boiling is resumed for 5 minutes, after which a filtration is made over a gaged 250 cc pyrex phial.

Washing with boiling acidulated water is effected until the water closely corresponds to the gage mark. The filtrate is preserved. (When the filtrate is cool, 10 cc is removed for titanium determination. The remaining 240 cc of filtrate will be used for iron determination).

The filter containing the silica is not dried. It is introduced directly in a platinum crucible and calcinated at a heat of 1,000° centigrade, in an electric oven or on a gas oven. Calcination is continued for 30 minutes after the filter-paper has been burned to ashes.

The crucibles are left to cool on triangles, and are then placed in the dessicator when come to a temperature of about 40° centigrade.

After cooling, the crucible is weighed a first time, defining weight A_1 g.

Eight drops of 1/1 sulfuric acid are then added, and the crucible is half filled with hydrofloric acid. Evaporation is ensured by slow heating on a sand bath.

When the crucible is dry, gas calcination is effected again for 30 minutes to ensure evaporation of the silica in the form of volatile silicon fluoride.

The crucible is cooled in the dessicator. It is weighed again, showing weight A_2 g. The difference is the weight of the silica contained in the sample.

/We have

we have: $A_1 - A_2 \times 100 = SiO_2$ %

(b) Determination of Fe_2O_3

Iron is determinated by means of a solution of potassium bichromate in presence of diphenylamine or barium diphenylaminosulfonate as an indicator.

Stannous solution:

5 g Cl_2Sn in 100 cc ClH H_2O 1/1

Standard potassium bichromate solution, 5 g per litre.

Diphenylamine indicator:

1 g for 100 cc SO_4H_2

or barium diphenylaminosulfonate:

0.5 g for 100 cc H_2O

To the filtrate obtained in the determination of total silica content (after removing 10 cc of that filtrate for titanium determination) we add without heating to the remaining 240 cc of filtrate previously poured into an erlenmeyer flask, a few drops of MnO_4K at approximately N/10, until a permanent pink hue is obtained to destroy organic matter.

Then heat to approximately 50° centigrade and add 10 cc of concentrated ClH.

Heat is increased almost to boiling point, the liquid turns yellow.

Reduction is effected with a few drops of Cl_2Sn, and is completed when the liquid becomes colourless.

As the samples become discoloured, they are cooled to room temperature by immersion in a container of cold water.

After cooling, add:

- 10 cc of SO_4H_2 at 1/6
- 10 cc of PO_4H_3 at 1/3
- 10 cc of Cl_2Hg at 50 g per 1,000 cc

a white turbidity should appear.

/Then add:

Then add:

- 2 cc of barium diphenylaminosulfonate, pour $Cr_2O_7K_2$ at 5 g per 1,000 cc until a violet colouring shows that the reaction is completed.

$$\frac{5.585 \times 5 \times 250 \times 100 \times 160}{4.90 \times 1000 \times 240 \times 112} = A$$

$A \times B = Fe_2C_3$ %

B = number of cc of bichromate

A = 0.849 for B = 1 cc.

The above computations should be effected once and for all; in fact, B is read on the burette, and referred to a correspondence table.

(c) Determination of TiO_2

Titanium determination is effected by the colorimetric method (e.g. by means of the Dubosq tintometer).

We had previously taken a sample of 10 cc of the solution obtained after silica filtration, and diluted to 250 cc, which are introduced in the tintometer tube.

Add:

5 cc of PO_4H_3

10 cc of SO_4H_2 at 1/3

and also, just before introducing in the tintometer:

2 cc of H_2O_2 at 20 volumes.

Comparison is made with a type sample of TiO_2 solution (1 g per litre) through addition of the same quantities of phosphoric acid, sulfuric acid and hydrogen peroxide, and the final volume of the sample under assay is obtained by addition of distilled water. We then have:

$$c' = \frac{c \cdot 1 \times 250 \times 100}{1' \times 10} = TiO_2 g$$

c' = unknown strength

c = standard known TiO_2 strength (in grams)

1 = distance traversed by the light of the standard solution.

1' = length traversed by the light of the solution of unknown strength.

/d) Determination

(d) Determination of Al_2O_3

Al_2O_3 can be obtained by substraction:

$$100 - (H_2O\% + SiO_2\% + Fe_2O_3\% + TiO_2\%) = Al_2O_3\%$$

Results:

Ignition loss	
Total SiO_2	
TiO_2	
Fe_2O_3	
Al_2O_3 by substraction	
Total	100%

Bangkok, 8 January 1962

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ANNEX 1

VARIOUS SOLUTIONS USED

Reaction solution

Distilled water	485 cc
Sulfuric acid	115 cc
Hydrochloric acid	300 cc
Nitric acid	100 cc

Stannous solution

Distilled water	1,000 cc
Chloric acid	1,000 cc
Chrystallized stannous chloride	100 g

Mercuric Chloride

Mercuric Chloride	50 g
Distilled water	1,000 cc

Barium diphenylaminosufonate

Barium diphenylaminousulfonate	5 g
Distilled water	1,000 cc
Hydrochloric acid	25 cc

Standard potassium bichromate

Potassium bichromate	5 g
Distilled water	1,000 cc

Sulfuric acid 1/2

Distilled water	500 cc
Sulfuric acid	500 cc

Sulfuric acid 1/3

Distilled water	650 cc
Sulfuric acid	350 cc

Sulfuric acid 1/6

Distilled water	1,000 cc
Sulfuric acid	200 cc

Phosphoric acid 1/3

Distilled water	650 cc
Phosphoric acid	350 cc

Acidulated water for washing

Distilled water	1,000 cc
Hydrochloric acid	10 cc

ANNEX 2

DETERMINATION DIAGRAM